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## A STUDY OF SHALLOW AND DEEP DAMAGE IN Cu AFTER IMPLANTATION OF 100 keV Cu AND Ag IONS

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In a recent study [1] we deduced the damage profiles in Cu and Al crystals due to self implantation from the measured dechanneling cross section. On the basis of this work a model for the observed shallow and deep damage was proposed.

In the present work this model is tested by comparing the damage in Cu crystals due to 100 keV self implantation at RT and due to 100 keV implantation with Ag ions at RT. In both cases regions of shallow and deep damage were found. The range of the shallow damage coincides approximately with the projected range of the implanted particles. The deep damage was found to extend to approximately  $6 \times$  this depth after Cu implantation and to approximately  $5 \times$  this depth after Ag implantation. After annealing the range of the deep damage was found to shrink. All these observations are in accordance with the proposed model.

### 1. Introduction

From earlier experiments [1] it was deduced that the damage formed by self implantation in copper and aluminium consists of two regions: a surface region coinciding with the expected depth distribution of the implanted ions and a deep region extending up to 10 times beyond this region. These experiments were performed using the channeling technique. With this technique the RBS yield  $\chi$  in a plane or string normalized with the random yield is determined in implanted ( $\chi_d$ ) and unimplanted (virgin) spots ( $\chi_v$ ). The values of  $\chi_d$  and  $\chi_v$  can be determined as a function of the depth in the crystal. Using the formula

$$I(z) = -\ln \left\{ \frac{1 - \chi_d(z)}{1 - \chi_v(z)} \right\} \quad (1)$$

a measure  $I(z)$  for the damage integrated from the surface to a depth  $z$  can be derived. The approximations used in the derivation of this formula are described elsewhere [1,2]. It has been shown [3] that this measure shows a linear dependence on  $\sqrt{E_1/Z_1}$  in case the damage is formed by dislocation lines, and no energy dependence if the damage consists of point defects, voids, clusters, bubbles or stacking faults.  $E_1$  and  $Z_1$  are the energy and atomic number of the analyzing particles.

In ref. 1 a microscopic model for the formation of shallow and deep damage is proposed, which is summarized in section 2.

In the present experiment we compare the results of channeling studies on damage by implantation of Ag and Cu ions in Cu with the proposed model.

### 2. Model

In ref. 1 a microscopic model has been proposed based on channeling work and other studies of damage by self implantation using transmission electron microscopy and field ion microscopy techniques and also simulation studies.

In this model the shallow damage is formed by depleted zones containing small vacancy clusters and (di)vacancies. This network of vacancy type of damage may collapse partly to form vacancy clusters or dislocation loops. The interstitial atoms are formed for the most part in a region separated by some nm from the depleted zone. The interstitials are highly mobile and diffuse to the surface and the depleted zone. It has been estimated that about 10% will escape from immediate recombination with vacancies. Using the modified Kinchin and Pease model [4] the number of created Frenkel pairs is about 200 per incident ion for implanta-

tion with 100 keV ions. With an implantation dose of  $2 \times 10^{20}$  ions/m<sup>2</sup> the number of "free" interstitials is of the order of  $4 \times 10^{21}$  per m<sup>2</sup>. Part of these interstitials will migrate into the crystal and aggregate due to their long range interaction and high binding energy to form small mobile clusters. These clusters may be pinned by an impurity or are lost at a dislocation or another sink. The aggregation is thought to be a crucial step in the process, because a single self-interstitial atom and an impurity do not form a stable complex at RT [5]. These pinned clusters will grow by capturing mobile (clusters of) interstitials forming the deep damage. Clusters containing more than 10 to 13 interstitials will convert to frank sessile loops [6]. A maximum damage will be reached if the loops overlap and a dislocation entanglement will result. The depth range in which the mechanism will work is limited because the concentration of mobile defects becomes too low for the fusion into larger clusters. This limit must be sharp since cluster formation must be proportional to  $C^n$  where  $C$  is the concentration of coagulating defects and  $n \geq 2$ . When the irradiation proceeds the growth of pinned clusters will slow down due to capture of interstitials by damage already created. This would imply that the deepest damage created at RT consists of small clusters which are immobile at RT. At prolonged irradiation, the depth, where the growth has been sufficient to create stable defects, will increase. This scheme of what may happen during irradiation yields a square damage profile and a range slightly increasing with the implantation dose as is observed in [1] and also in the work of Sood and Dearnaley [7].

On annealing, part of the pinned clusters may become mobile to form larger clusters or loops. At greater depth, where the concentration of damage is low, these clusters may escape without fusing. This would explain the annealing behaviour, including the loss of damage at greater depth.

### 3. Experimental procedure

Single crystals of high-purity (99.999%) Cu of  $\langle 110 \rangle$  normal orientation were electropolished. A part of the surface was implanted at RT with  $2 \times 10^{20}$  m<sup>-2</sup> Ag ions of 100 keV or with  $2 \times 10^{20}$  m<sup>-2</sup> Cu ions of 100 keV. An Ag-implanted sample was thermally annealed in a hydrogen atmosphere

at 625 K during 1 h. In this way 4 types of samples were prepared: unimplanted samples, Cu-implanted samples and Ag-implanted samples before and after annealing. Of each of the 4 samples RBS spectra were taken with cooled silicon surface-barrier detectors (15 keV fwhm resolution), placed around 180° and at 165° and 135° with respect to the beam direction. This was done at various beam energies with the beam aligned with the normal  $\langle 110 \rangle$  string, with a  $\langle 111 \rangle$  plane 7° from the normal string and in a random direction 3° from the  $\langle 111 \rangle$  plane. Each series of measurements at one energy was performed at a fresh spot on the crystal. Beams of proton and alpha particles with energies between 0.5 and 4.0 MeV from the Groningen 5 MV Van de Graaff accelerator were used. To perform the alignment the samples were mounted on a 2-axis goniometer provided with an  $x$ - $y$  translational system with which the sample could be moved with respect to the beam. The whole measurement was carried out under computer control.

### 4. Results

In fig. 1 a set of spectra normalized with a random spectrum is shown. The set was obtained by bombarding an Ag-implanted Cu crystal with 0.5 MeV  $\alpha$  particles. Basically the (111) spectrum displays  $\chi_d$  as a function of depth. With eq. (1) it can be understood that the slope of the spectrum is a rough measure for the local damage as a function of depth. The shallow damage, partly hidden by the surface peak, can be seen to extend to channel 600 which corresponds to a depth of 55 nm. The deep damage extends from channel 600 to channel 300, corresponding to a depth of 250 nm.

In fig. 2 the depth profile of the damage is displayed. It was obtained by applying the differential form of eq. (1) to the spectra of  $\chi_d$  and  $\chi_v$ . Before calculating eq. (1), these spectra were fitted by a spline function. In the figure the depth profile is compared with the measured depth profile of the implanted Ag as obtained from the part of a spectrum obtained with 4 MeV  $\alpha$  particles shown in fig. 3. Due to energy straggling the damage profile as well as the Ag profile are smeared out at the deep side. However, it is without doubt that the damage extends much further than the range of the Ag.

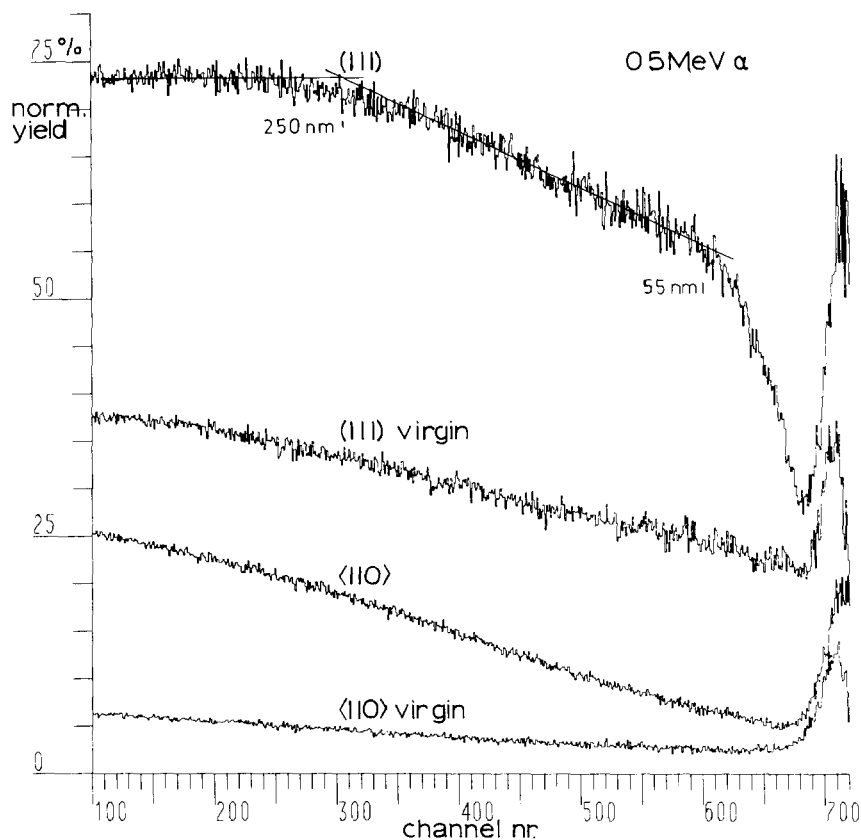


Fig. 1. Spectra obtained with 0.5 MeV alpha particles in the  $\langle 110 \rangle$  and  $\langle 111 \rangle$  direction normalized by a random spectrum in Ag-implanted and virgin Cu.

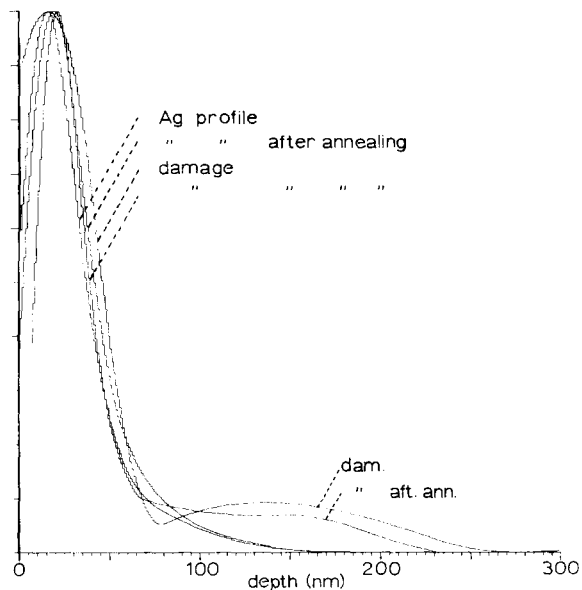


Fig. 2. Depth distribution profile of Ag obtained from the 4.0 MeV alpha spectra and the damage profile  $D(z)$  obtained from the 0.5 MeV alpha spectra, before and after annealing.

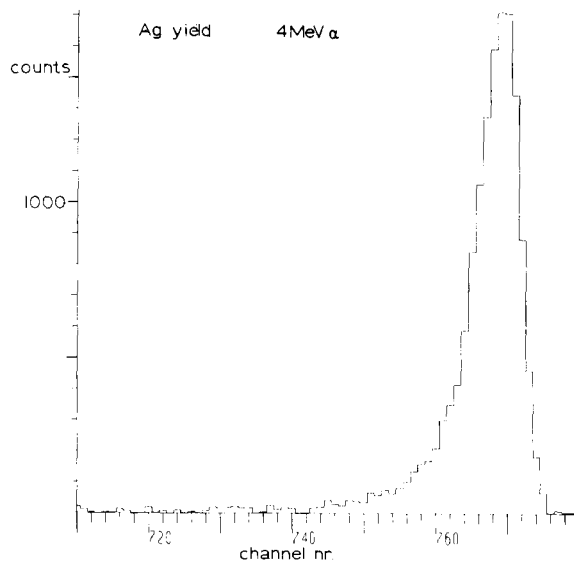


Fig. 3. Part of the spectrum of 4 MeV alpha particles showing the Ag peak.

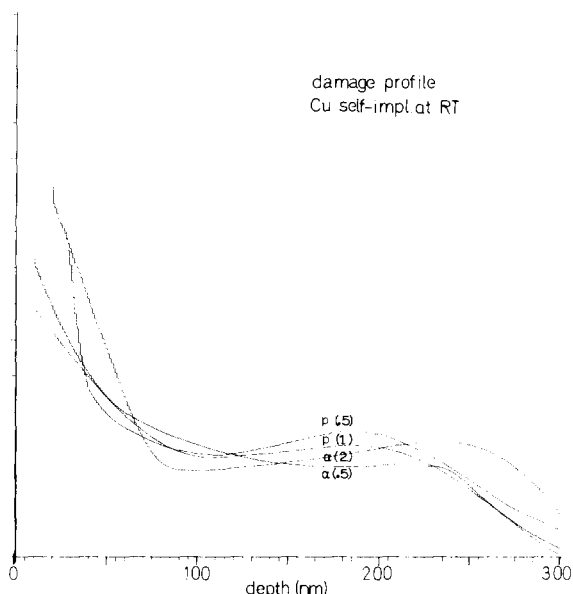


Fig. 4. The damage profile  $D(z)$  for copper self-implanted at RT. The particle type and energy are indicated with  $\alpha(E_1)$  for alpha particles and  $p(E_1)$  for protons.

In fig. 2 we also compare the depth profiles for Ag atoms and the damage, before and after annealing. It is clear that the deepest damage has disappeared and that the Ag profile has become broader.

In fig. 4 results for a self-implanted copper crystal are given. The depth profile of the damage as obtained with different particles and energies is shown. Also here we can distinguish a shallow and a deep region of damage. The deep region extends to about 300 nm, decreasing to about 250 nm after annealing at 625 K.

## 5. Discussion of results

From damage profiles (see fig. 4) it is evident, that the damage induced in a single copper crystal by self implantation with 100 keV Cu ions, consists of two regions: a shallow region coinciding with the expected profile of the implanted ions, and a deep region extending to a depth of 300 nm. The approximately square profile of the deep damage is in agreement with the predictions of the model.

In terms of the model the depth of the deep damage is mainly determined by the number of nucleation centres for interstitial clusters. The

copper of the single crystals contains an atomic fraction of  $10^{-7}$  Ag atoms. It has been shown in [1] that this fraction may explain the observed depth.

The damage induced by Ag-implantation in the Cu single crystal is of similar character. The shallow damage coincides approximately with the depth profile of the implanted Ag (fig. 2); the deep damage extends to 250 nm in this case instead of 300 nm in the case of Cu implanted copper.

Again this can be understood on the basis of the model: the increased Ag concentration due to the implantation provides a higher density of nucleation centres. This leads to more effective screening of deeper parts from interstitials. As in the case of Cu-implanted Cu the range of the deep damage shrinks by 40 nm after annealing at a temperature of 625 K for one hour. In terms of the model this can be understood by the release of small interstitial clusters, just bound to their nucleation centres at RT. A small, but significant increase in the depth of the implanted Ag is observed (see fig. 2). This increase may be caused by damage-assisted diffusion due to the break-up of small vacancy clusters in the shallow region. Thermal diffusion is negligible at 625 K.

## 6. Conclusions

Similar shallow and deep damage regions were found after implantation of Cu single crystals with Ag or Cu ions of 100 keV. The range of the deep damage including the differences in ranges for Ag and Cu implantation may be understood in terms of a model, which also explains the anneal behaviour.

A precise quantitative verification of the model by solving the relevant diffusion equations is impossible as long as the parameters needed, such as capture radii and binding energies for (clusters of) interstitials, are unknown.

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